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LA-UR--84-1950

DE84 014040

TITLE. DEVELOPMENT AND EXTENDED OPERATION OF A HIGH POWER RADIATION  
LOADED HEAT PIPE

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SUBMITTED TO: 19th AIAA Thermophysics Conference  
Snowmass, Colorado  
June 24-28, 1984

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7/1/84

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# **AIAA'84**

**AIAA-84-1719**

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## DEVELOPMENT AND EXTENDED OPERATION OF A HIGH POWER RADIATION LOADED HEAT PIPE

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### Abstract

A high temperature, high power molybdenum-lithium heat pipe has been fabricated and tested at 1500 K for 1700 hours with radiant heat rejection. Power throughput during the test was approximately 14 kW, corresponding to an axial flux density of 11 kW/cm<sup>2</sup> for the 1.59 cm. diameter heat pipe. Radial flux density was 70 W/cm<sup>2</sup> over an evaporator length of 40.0 cm. Condenser length was approximately 150 cm. with radiant heat rejection from the condenser to a coaxial water cooled radiation calorimeter. A plasma sprayed, high emissivity coating was used on the condenser surface to increase the radiant heat rejection during the tests. The heat pipe was operated for 514 hours at steady state conditions before being damaged during a planned shutdown for test equipment maintenance. The damage was repaired and the initial 1000 hour test period completed without further incident. After physical examination of the heat pipe at 1000 hours the test was resumed and the heat pipe operated at the same conditions for an additional 700 hours before conclusion of this test phase.

### Introduction

High power, high temperature heat pipes have been proposed for use in space power systems where reliability of operation is of prime importance. Demonstration of the operation of such heat pipes over periods of thousands of hours under realistic test conditions has yet to be accomplished. Life test heat pipes have been operated for periods of greater than 20,000 hours at temperatures of 1500 K to 1700 K<sup>1</sup> but without high power throughput. The complexity of testing at combined high temperature and power has discouraged life testing prototypical hardware. Tests have been conducted for periods of hundreds of hours at axial power density levels in excess of 10 kW/cm<sup>2</sup> with condenser coupling controlled by means of gas-gap calorimeters,<sup>2</sup> however even for these shorter duration tests inherently low emissivity of the refractory materials used for the heat pipe envelope has limited radiant heat rejection. This mode of condenser heat rejection is of interest because it is the mode of choice in many space power applications. For systems employing thermoelectric energy conversion the use of radiation coupling provides a practical alternative to high temperature electrical insulation at the energy input side of the thermoelectric elements. Prior test program developments at Los Alamos have demonstrated the feasibility of using plasma sprayed zirconium

diboride coatings on molybdenum heat pipes as a method of attaining surface emissivities greater than 0.6.<sup>3</sup> This allows the achievement of power throughput levels of 12 to 15 kW with total heat pipe lengths of about 2 m. The present investigation was planned to provide a demonstration of an operation of the high temperature heat pipes at high radial and axial heat flux with radiation loading of the condenser.

### Method of Approach

A 2.0 m long, annular wick heat pipe capable of transporting an axial flux of greater than 10 kW/cm<sup>2</sup> was designed and fabricated for use in an extended test program at a nominal operating temperature of 1500 K. The design employed molybdenum alloys for screen wick materials and for the tube shell, with lithium as the working fluid. The condenser region of the heat pipe was coated to increase the effective hemispherical emissivity of the surface. Use of the high emissivity coating gave a predicted test throughput for the heat pipe of 14 kW at 1500 K with radiation coupling to an ambient temperature calorimeter. A test period of 1000 hours at this operating condition was the design goal for the program.

### Test Hardware

The 2 m long heat pipe was fabricated from 1.59 cm. outside diameter low carbon arc cast molybdenum tubing having a wall thickness of 0.89 mm. End closures were machined from bar stock of the same material. A cross section of the heat pipe is given in Fig. 1.

The screen wick was fabricated from a single piece of 400 mesh plain square weave wire cloth woven from 0.025 mm diameter molybdenum -41% rhenium wire. In fabricating the wick the screen was wrapped on a low carbon steel tube mandrel to form a structure 6.75 layers thick. A series of spot weld along the leading and trailing edges of the wrap were used to hold them in place during forming and processing. A sheath, also of low carbon steel, was fitted over the screen layers and the assembly drawn through a sizing die to compress the layers. The reduction in screen cross section was approximately 20%. The steel mandrel and sheath were then removed by dissolution with hydrochloric acid to leave a clean cylindrical wick. The wick was further treated by vacuum firing at 1585 K for 5 hours in order to sinter the screen layers into a monolithic structure and to remove any volatile contaminants.

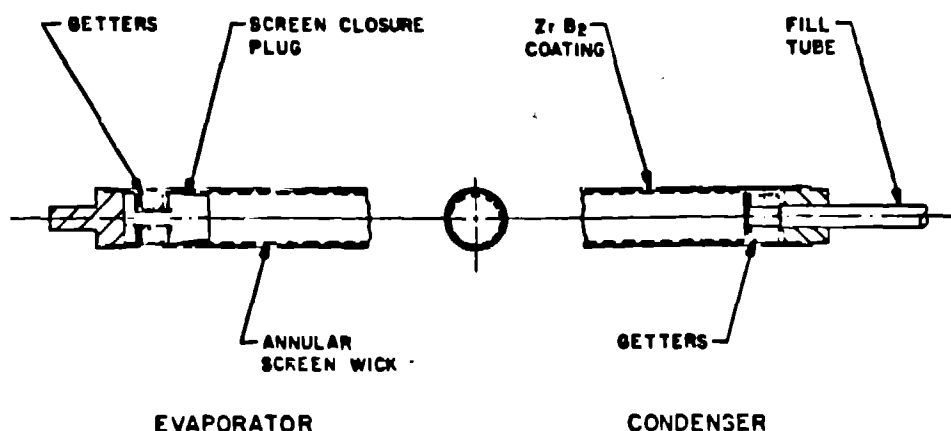


Fig. 1. Cross section of extended operation test heat pipe.

Surface tension measurements of the completed wick indicated an effective pore diameter of less than 49 microns and a permeability of  $2.5 \times 10^{-12} \text{ m}^2$ , within the required design tolerance. The fluid annulus, which extended the length of the heat pipe, was formed between the container tube inside diameter and the slightly smaller tubular wick. This annulus was intended to serve as an artery for fluid return from the condenser to the evaporator during operation. A molybdenum plug was used to close the screen wick tube at the evaporator end. This plug closure ensured that the annulus pumping capability was established by the effective pore size of the screen wick rather than by the annulus dimensions. The wick outside diameter of 13 mm established a maximum dimension for the annulus of 0.86 mm when assembled in the 14 mm inside diameter container tube. The cross sectional flow area of the resulting annulus was  $0.19 \text{ mm}^2$ . Vapor flow area for the heat pipe based on the screen wick tube inside diameter was  $1.26 \text{ mm}^2$ .

The evaporator end of the screen wick was sealed with a molybdenum plug using a vacuum braze joint made with preplaced zirconium wire braze material. Two turns of 0.381 mm diameter zirconium wire were pressed into the wick. Surface tension measurements of sample braze closures were used to establish the integrity of the braze plug closure process. It was found that at brazing temperature the capillary forces in the wick would move the zirconium braze material away from the joint interface unless an interference fit of 0.051 mm was maintained between the plug and the bore of the screen wick tube. Brazing was accomplished by RF induction heating the joint in a quartz envelope that allowed visual observation of the braze material. In making the joint the RF power was slowly increased until the braze was observed to flow. The power and temperature were then immediately reduced and the joint brought to ambient temperature over a period of approximately one-half hour.

Hafnium and zirconium disks were placed inside each end of the heat pipe to serve as getters for carbon, nitrogen and other trace contaminants during the operation.

Closure of the heat pipe envelope was accomplished by electron beam welding a fill plug assembly in place after wick insertion. The fill plug assembly consisted of the tube closure pre-assembled to a fill tube of 6.35 mm molybdenum tubing with a niobium braze transition to stainless steel for fill system attachment. After completion of the weld the system was leak checked with a  $9 \times 10^{-11} \text{ atm cm}^3/\text{sec}$  leak detector. No leaks were disclosed. Prior to filling the heat pipe assembly was further checked by radiographic examination with no evidence of flaws. Following this examination the heat pipe was processed for increased emissivity of the outer surface over all but the evaporator region. This was accomplished by bead blasting the outer surface of the LCAC molybdenum tube and then DC plasma arc spraying zirconium diboride on the surface. Film thickness of the coating was approximately 0.08 mm with a coating density of about 85% of theoretical. An argon carrier gas was used for the plasma spray process.

#### Distillation and Pre-test Processing

The distillation apparatus used to charge the heat pipe with lithium is shown schematically in Fig. 2. The stainless steel distillation pot contained multiple layers of 100 mesh stainless screen wrapped next to the inside surface to provide an extended area of surface for lithium evaporation. In operation the distillation pot was RF induction heated at a level just above the surface of the liquid metal pool. Transfer of fluid to the evaporation surface was accomplished by capillary action through these screen layers. Carryover of masses of liquid through slugging or extremely active boiling was thereby eliminated. Hafnium and zirconium getter materials were used to purify the lithium during the distillation process. The getter materials were positioned in the apparatus so as to be completely immersed in the liquid lithium pool during distillation. The fill volume chamber was attached to the loading pot by a stainless steel tube that extended through the liquid pool. This tube provided a means to evacuate the heat pipe during the degassing procedure prior to distillation and served as a path for the lithium vapor during the

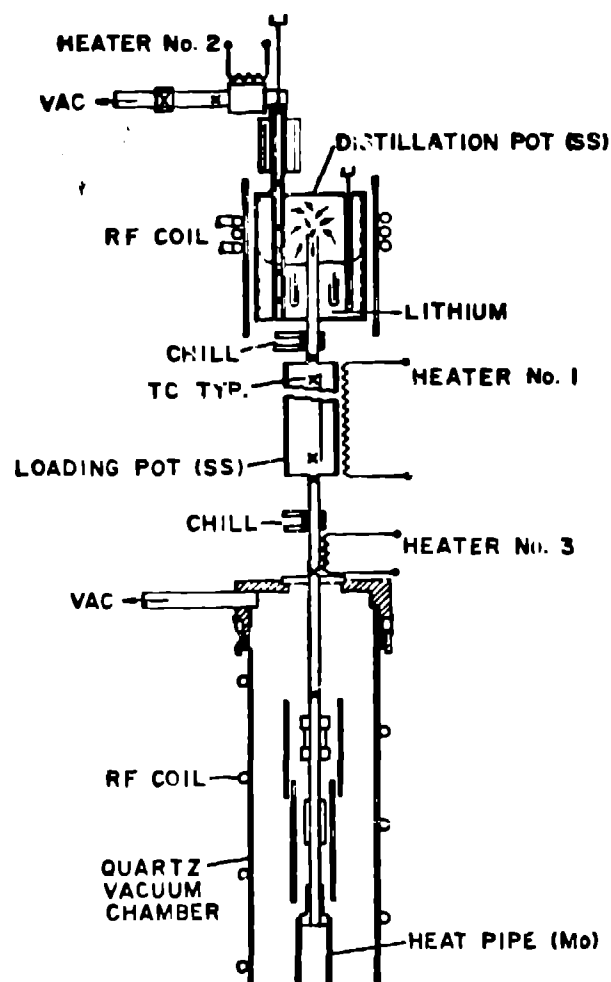


Fig. 2. Lithium distillation fill assembly.

fill process. A secondary transfer tube extended from the fill volume chamber to the heat pipe. A water cooled chill block mounted on this tube was used as a valve during the filling operation. This freeze valve was used to prevent molten lithium from draining out of the chamber during distillation. Both the fill tube and the loading pot were sized to provide a volume of lithium sufficient to fill the wick structure of the heat pipe and to provide the desired pool length of surplus lithium within the heat pipe. The heat pipe internal volume was connected to a vacuum pumping system through the distillation lines. This vacuum system was used for degassing the heat pipe interior during the pre-fill vacuum bakeout and also for degassing the lithium working fluid charge during warm-up, initial lithium melting, and distillation. The vacuum line was kept open during the distillation by a temperature controlled liquid trap in the pump line. Lithium vapor in the vacuum pump line was condensed in this water cooled trap and returned to the distillation pot by capillary action through the screen wick liner. This open system design allowed for the monitoring and removal of non-condensable gases evolved during the distillation. A residual gas partial pressure analyser

attached to the vacuum pump line was used to monitor the off gases throughout the distillation process. During the vacuum bakeout and distillation process the heat pipe exterior was protected from oxidation by the surrounding quartz envelope. The envelope was connected to a second pumping system and maintained under vacuum throughout the processing to prevent oxidation of the heat pipe exterior and possible contamination of the heat pipe with hydrogen due to disassociation of moisture on the heated surface. Heat pipe degassing and filling was conducted with the heat pipe oriented vertically. The entire heat pipe was first brought to a temperature of 1500 K using a full length RF coil. The rate of warm-up was controlled to maintain the pressure in the interior and exterior vacuum systems below  $10^{-4}$  torr throughout the process.

The fill lines, loading pot and distillation pot were subsequently heated at a controlled rate to maintain the same vacuum levels. The loading and distillation pots were held at 1050 K until the inner system vacuum levels reached  $10^{-6}$  torr. The loading pot was then cooled to 500 K, the lower fill line and heat pipe cooled to 300 K and the lower chill activated. The distillation pot temperature was then increased to 1150 K and the purified lithium vapor condensed into the loading pot. The volume from the chilled heat sink to the top of the transfer tube was filled with lithium to complete the distillation. The distillation pot was cooled to 600 K and the heat sink removed. The heat pipe, transfer tube, and loading chamber were then heated to 500 K allowing the lithium to transfer into the heat pipe under combined pressure differential and gravity head. The system was then cooled and the heat pipe and distillation apparatus positioned in a horizontal orientation. For wet-in the heat pipe was heated with a full length RF coil to 1100 K and maintained at this temperature overnight. The wet-in operation served to distribute the lithium charge uniformly throughout the heat pipe and to ensure uniform wetting of all surfaces.

For initial performance verification the full length coil was replaced with a 30 cm long coil. Length of the excess liquid pool at the condenser end of the heat pipe was determined at the same time. The heat pipe was then operated off horizontal, that is with a gravity head, for an additional period to ensure wet-in. The distillation apparatus was then removed and the fill line capped. Operation of the heat pipe prior to fill line separation was such as to ensure the presence of a frozen plug of lithium in the fill tube. After capping, a chill block (water cooled) was positioned on the fill line and clamped in place. This heat sink was maintained below 350 K in subsequent heat pipe operation. Final processing of the heat pipe prior to coating involved positioning a 30 cm coil at the evaporator end of the heat pipe and operating in a vertical orientation with adverse gravity head. (The evaporator at the top). A temperature of 1500 K was reached in operation with a corresponding heat throughput of approximately 4 kW due to thermal radiation loading. Following this performance verification the heat pipe was installed in the radiation calorimeter and instrumented for the extended operation test.

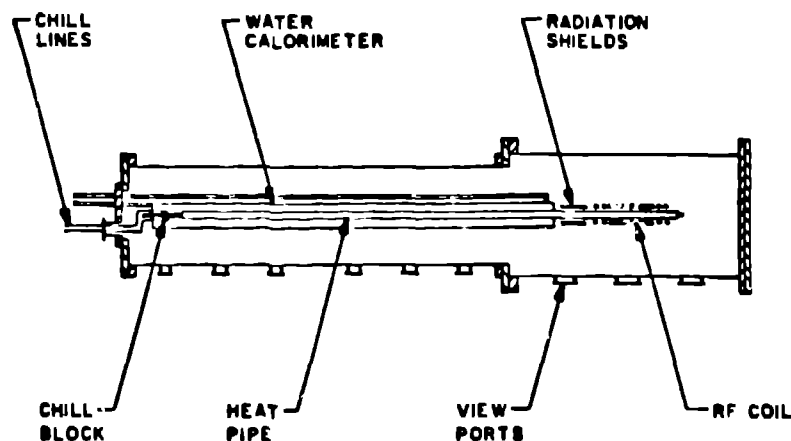


Fig. 3. Heat pipe test arrangement.

### Test Operation

Power input to the heat pipe during the extended operation test was provided by RF induction at approximately 400 kHz, and a water cooled coaxial calorimeter served as the heat sink for radiation from the condenser as indicated in Fig. 3. Power throughout was determined from water flow and temperature change measurements at the calorimeter. Heat pipe temperature was measured by tungsten-rhenium thermocouples welded to the heat pipe in the evaporator exit area. The test was conducted in a vacuum chamber maintained at a pressure level of  $10^{-6}$  torr throughout the test.

After filling and wet-in of the heat pipe it was installed in the test chamber and operated at 1400 K for 6 hours for operational verification. The test was then shutdown over a weekend. The following Monday the heat pipe was brought to the extended operation test condition by increasing the RF power to the evaporator over a period of hours. An axial temperature profile of the condenser region of the heat pipe was taken by optical pyrometry as a measure of start-of-test performance. The measured temperature variation over the length of the zirconium-diboride coated region of the heat pipe was 8 K. The effective spectral emissivity of the coating at the detection wavelength of the optical pyrometer was determined by comparison of the measured temperature with known values for cleaned molybdenum. The comparative measurements were taken adjacent to each other at a location on the heat pipe near the evaporator exit. Once the design temperature of 1500 K had been achieved the heat pipe was operated at constant power input. Steady state operation was continued for a period of 514 hours with no apparent change in the heat pipe operating characteristics. At 514 hours the test operation was shut down for planned maintenance of the RF heating system. In the process of reducing the power and temperature of the heat pipe a dry-out of the heat pipe evaporator occurred. Restart attempts led to repeated evaporator dry outs.

The heat pipe was maintained overnight at 1075 K in an attempt to rewet the dried out region of the evaporator without success. The heat pipe was removed from the test chamber for X-ray

and neutron radiography to determine the condition of the wick structure and the disposition of the lithium in the annulus. The radiography showed transfer of lithium to the condenser end of the heat pipe leaving the annulus empty in the evaporator region. The X-rays indicated damage to the wick structure in the evaporator. To effect a repair the evaporator end of the heat pipe was opened, the screen tube shortened by 10 cm, and the evaporator end of the screen wick resealed with a brazed plug. The annular screen wick was reinserted into the shortened container tube, new getter discs of hafnium and zirconium emplaced outside of the screen wick end plug and a tube end closure electron beam welded in place to reseal the heat pipe. The repaired heat pipe was refilled with lithium, wet-in by operation at low power in multifoil radiation shields and reinstalled in the test chamber.

The test was restarted and the heat pipe operated continuously for an additional 500 hours at 1500 K and 14 kW to complete the originally planned 1000 hour demonstration. At the conclusion of 1014 hours of operation of the heat pipe the optical axial temperature profile of the condenser region was repeated to evaluate change in the heat pipe operating characteristics or in the zirconium-diboride optical properties. Based on the relative spectral emissivity value determined at the beginning of test no change was detected. At the completion of the test period the heat pipe was removed from the test chamber for a repeat of X-ray and neutron radiography and for general physical inspection. No evidence of further damage to the heat pipe structure was discovered in these examinations. However a void in the lithium fill of the annulus was observed in the evaporator region of the heat pipe.

The heat pipe was then rewet at 675 K for 5 hours in a vacuum furnace, reinstalled in the test chamber, brought to the design operating condition and operated for an additional 744 hours at 1500 K and 14 kW. At this time the test was interrupted by an unplanned transient in the data recording system which triggered an interlock shutdown of the RF generator. The heat pipe was allowed to cool, removed from the chamber and rewet in a vacuum furnace for 5 hours at 675 K. It was then reinstalled in the test chamber and brought to 1210 K and a power level of about

4.0 kW before the operation was interrupted by a vacuum interlock shutdown. Attempts to restart the heat pipe were unsuccessful. The heat pipe was then removed from the test chamber, and subjected to neutron radiography and X-ray examination. The radiographs again showed a lithium void in the evaporator annulus, however, the wick did not appear to be damaged. A decision was made at this point to discontinue the extended operation test and investigate the dry out conditions.<sup>4</sup>

#### Interpretation of Test Results

Prior to the start of the 1000 hour test the heat pipe was operated at 1400 K with the radiation calorimeter in the test chamber, shutdown, and restarted without incident. The initial dry-out occurred under power and near the sonic limit while reducing the heat pipe temperature after 514 hours of full power operation. This suggests that some deterioration of the wick structure leading to an increased capillary pore radius may have occurred in the first 500 hours of operation. If the wick effective pore size was unchanged at about 50  $\mu\text{m}$  the heat pipe would not have been expected to dry out due to evaporator subcooling. Any change in wick characteristics prior to the 500 hour dry-out would have had to be minor, however, the heat pipe continued to operate at full power and temperature, indicating an effective pore size of  $\sim 120$  to  $150 \mu\text{m}$ . The gross damage observed in disassembly at 514 hours must have occurred after normal operation had been interrupted.

Pending further investigation it is assumed that the cause of the restart problem at 514 hours was the formation of voids in the evaporator region of the annulus with local de-wetting of the heat pipe wick during fluid solidification. This is assumed to be caused by directional solidification of the heat pipe working fluid and is perhaps aggravated by an increase in the effective pore size of the heat pipe wick over the operating period. The dry out observed during shutdown under power may have been similarly caused by a change in the wick structure in the prior 514 hours of full power operation. At this point there is no evidence that operational interruption of the heat pipe occurs at the sonic limit point if the shut down is conducted by shutting off the input power.

#### Conclusions

The zirconium diboride coating showed no signs of degradation during the 1000 hour test. Optical properties appeared constant and the

coating to substrate adhesion integrity was maintained throughout both the experiment and the reprocessing of the heat pipe. Although the hemispherical emissivity of the coating is lower than that desired for use in prototypical radiation coupled systems its durability and ease of application warrant its use in high temperature radiation coupled heat pipe experiments. It is significant that the test heat pipe operated with no evidence of degradation for more than 1000 hours with an axial power density of  $11 \text{ kW/cm}^2$  at a temperature of 1500 K but was severely damaged by a brief period of operation at a lower temperature with a de-wetted evaporator region. Analysis indicates that molybdenum and rhenium oxides cannot be formed in the presence of liquid lithium, even with oxygen levels to  $\text{Li}_2\text{O}$ . However, in the de-wetted region of the evaporator, with no liquid lithium present, it might be possible to oxidize the screen wick material with subsequent reduction of the oxides by liquid lithium heading to the eroded wick structure seen in these experiments. Local dryout of this sort would be more probable in an RF induction heated heat pipe such as was used in these experiments than in one employing a temperature dependent heat source. However, for lithium heat pipes it is apparent that de-wetted evaporator conditions must be avoided if long operating life is to be achieved.

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